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An Optical Fibre Sensor of the Concentration of Urea Solutions

J.P. Legendre

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ABSTRACT

An investigation was undertaken to determine the feasibility of using optical fibre reflectometers as continuous monitors of the concentration of urea in water. The tests indicated that fibre reflectometers were able to detect the presence of very weak concentrations of urea in water (as little as 3 g/L) and were able to clearly distinguish between solutions whose concentrations differed very little (again, by as little as 3 g/L). The tests showed the expected correlation between reflectometer responses and refractive indices of the solutions.

RÉSUMÉ

Une étude de faisabilité a été entreprise afin de déterminer si un réflectomètre à fibres optiques pourrait servir comme moniteur continu de la concentration d'urée dans l'eau. Les essais ont montré que ces réflectomètres permettent de déceler des concentrations très faibles d'urée dans l'eau (aussi peu que 3 g/L) et de percevoir de très petites variations de concentration (là encore, aussi peu que 3 g/L). Ils ont aussi permis d'observer la corrélation prévue entre la réponse de l'appareil et l'indice de réfraction de la solution dosée.

AN OPTICAL FIBRE SENSOR OF THE CONCENTRATION OF UREA SOLUTIONS

J.P. Legendre

INTRODUCTION

An investigation was undertaken to determine the feasability of using an optical fibre reflectometer developed within the Division of Electrical Engineering as a continuous monitor of the concentration of urea in water.

Technology for producing sulphur coated urea (SCU) is being developed by Meisen and his colleagues at the Department of Chemical Engineering of the University of British Columbia. Sulphur coated urea is a controlled release fertilizer. In order to characterize the product, as well as control its manufacture, it is necessary to know its dissolution rate.

At present, standard tests require that a known quantity of SCU be placed in a volume of water for extended periods (typically 7 days). After this time, a sample of the water is tested to determine the concentration of urea present. Accelerated tests are also being developed. In either case, the concentration of urea which has leached through the sulphur coating and into the water must be determined. It is possible to determine urea concentration in water by measuring the refractive index of the solution. At the moment, this measurement is time consuming as it requires that a sample be placed in an instrument such as an Abbé refractometer.

It is the object of the present tests to determine if a continuous monitor of refractive index, placed in the water bath, might be practical.

PROCEDURE

Van Q. Le of the University of British Columbia prepared 10 samples for testing. Six of the samples (#1 - #6) were of known concentration. The remaining four samples (#7 - #10) were prepared by leaching SCU in a water bath for different periods of time. The refractive index of all 10 samples was measured with an Abbé refractometer. The samples along with the relevant data are listed in Table 1. The samples were then forwarded to the NRC.

One of the fibre optic reflectometers used in these tests has been described elsewhere (1). A schematic of this device, which uses a LED emitting 840 nm radiation, is presented in Fig. 1. Tests were also

conducted with a dual-wavelength reflectometer which is shown in Fig. 2. In this device, one LED emitted 840 nm radiation while the other emitted at 1272 nm. The LEDs were both square-wave modulated but at different frequencies. In this way, a single photo-diode could be used to measure the radiation reflected from the fibre tip simultaneously at two wavelengths and yet the information contained at each wavelength could be separated by using phase-sensitive detectors tuned to the different modulation frequencies.

Table 1

Samples	Refractive Index n	Concentration (g/L)
1	1.3457	100
2	1.3389	50
3	1.3357	25
4	1.3336	12.5
5	1.3332	6.25
6	1.3330	3.125
7	1.3420	
8	1.3425	
9	1.3435	
10	1.3442	

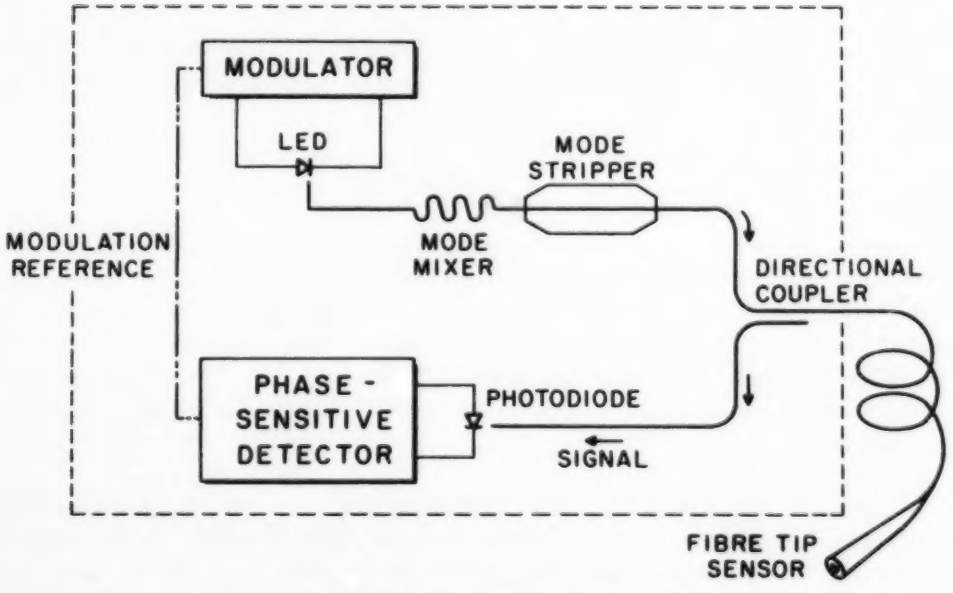

Figure 1 Single wavelength reflectometer schematic.

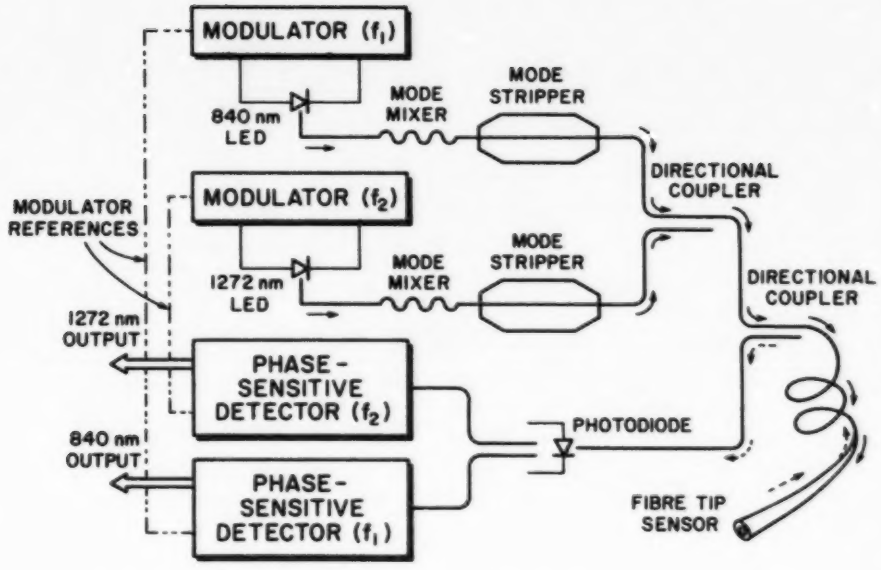

Figure 2 Dual wavelength reflectometer schematic.

The measurement procedure consisted, very simply, of presenting the different samples, in turn, to a fibre tip. Data acquisition proceeded while the fibre tip was thus immersed in a static solution. Between each sample measurement, the fibre tip was immersed in a static sample of distilled water. This served to rinse the fibre tip of any residue from the preceding solution sample and thereby minimize cross-contamination. It also served to re-establish a reference baseline signal to which the signals of the various solutions could be compared. Prior to starting any data recording, all solutions, including the reference sample of distilled water, were allowed to come to room temperature. During all reflectometer measurements, the room temperature in the immediate vicinity of the experiment was monitored with a thermocouple and recorded continuously.

The digital data acquisition system caused 1000 data points to be stored during the recording period for each channel. In the case of the dual wavelength reflectometer three channels were used, one for each of the wavelengths and one for the local temperature.

RESULTS

Figures 3 - 6

All data shown in these figures was acquired using the dual wavelength reflectometer discussed above. In each case the lock-in amplifier (phase-sensitive detector) time-constant was set at 125 ms. Figures 3 and

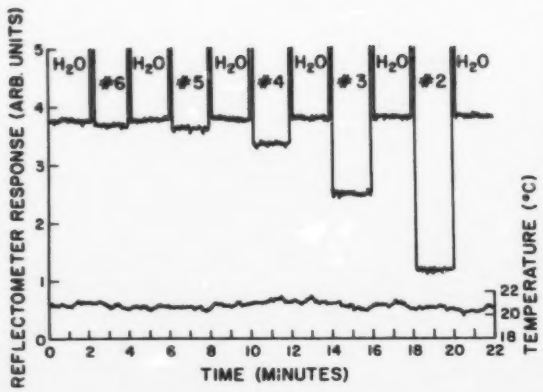

Figure 3 Response of 840 nm channel of the dual-wavelength reflectometer to various concentrations (samples 2, 3, 4, 5, 6 - Table 1) of urea in water. The probe tip was immersed in distilled water between each immersion in a test solution. Amplifier time constant was 125 ms.

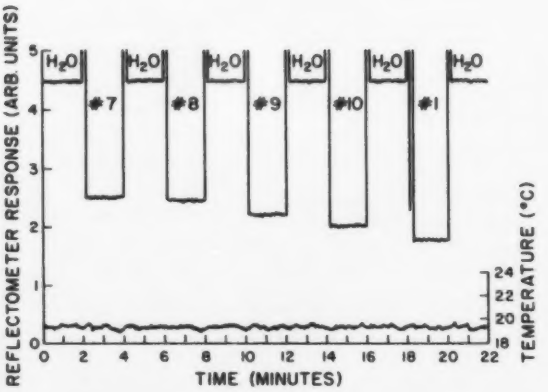

Figure 4 Response of 840 nm channel of the dual-wavelength reflectometer to various concentrations (samples 1, 7, 8, 9, 10 - Table 1) of urea in water. The probe tip was immersed in distilled water between each immersion in a test solution. Amplifier time constant was 125 ms.

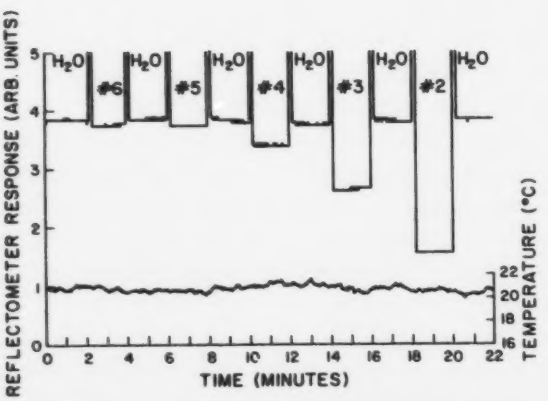

Figure 5 Response of 1272 nm channel of the dual-wavelength reflectometer to various concentrations (samples 2, 3, 4, 5, 6 - Table 1) of urea in water. The probe tip was immersed in distilled water between each immersion in a test solution. Amplifier time constant was 125 ms.

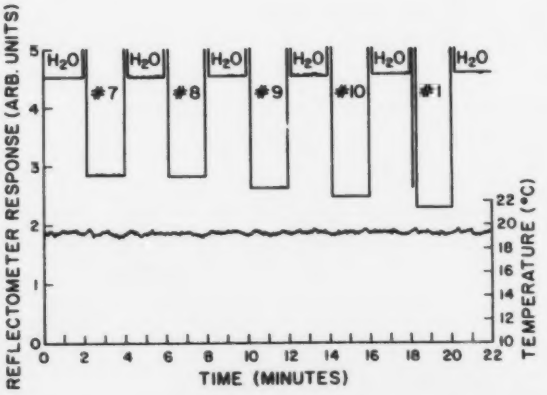

Figure 6 Response of 1272 nm channel of the dual-wavelength reflectometer to various concentrations (samples 1, 7, 8, 9, 10 - Table 1) of urea in water. The probe tip was immersed in distilled water between each immersion in a test solution. Amplifier time constant was 125 ms.

4 show the response of the reflectometer at 840 nm while Figs. 5 and 6 give the response at 1272 nm. In most cases, the samples are clearly distinguished one from the other by the signal responses illicited. The only exception to this are the responses to samples #6 and #5 shown in Fig. 5. In all cases, including those samples containing the weakest solutions, the sample responses are very clearly distinguished from the response to the reference sample of pure water. The data show that the reference levels were very constant with no evidence of signal drift.

Figures 7 - 8

Data shown in these figures were acquired using a single wavelength reflectometer operating at 840 nm. The lock-in amplifier time-constant for these trials was set at 1.25 ms. The only effect which the use of this inappropriate time constant should have is to make the data appear noisier than would normally be the case since a much shorter integration time was effectively used to record each data point. These records show that all samples are distinguished from one another and from the reference water sample by the signal responses which they illicit. There are, however, some aspects of these results which distinguish them from those obtained with the dual-wavelength unit discussed above. Perhaps least significant is the drift in the reference signal levels. This reflectometer is an older unit and is known to have inadequate cooling of critical electronic components. More curious is the appearance of the response, as it varies in time for the various samples, in Fig. 8. In each case the response appears to be approaching some final value asymptotically. The variation of the signals from the water reference were initially thought to be due to local heating of the water sample by the radiation from the LED. It is difficult to sustain this view when Fig. 7 is examined - no such asymptotic variation is evident there. It is also curious that the signal variations for the numbered samples (with one exception) are in the opposite sense to those signal variations due to the water reference. The one exception in Fig. 8 is the signal response due to sample #1 which shows little of the asymptotic variation evident elsewhere.

Is it significant that the samples which yield anomalous results (i.e., samples 7, 8, 9, and 10) were all prepared from sulphur coated urea? Is it possible that these solutions might contain some contaminant, chemical or bacteriological, which might explain the results? I also have no explanation for the fact that the dual wavelength reflectometer did not show similar signal variations for these identical samples at either wavelength other than to suggest that some form of contamination was present in one case and not in the other. It should be stated that the curious results were present in several trials. Between each trial the fibre tip was examined and cleaned, if necessary. Past experience has

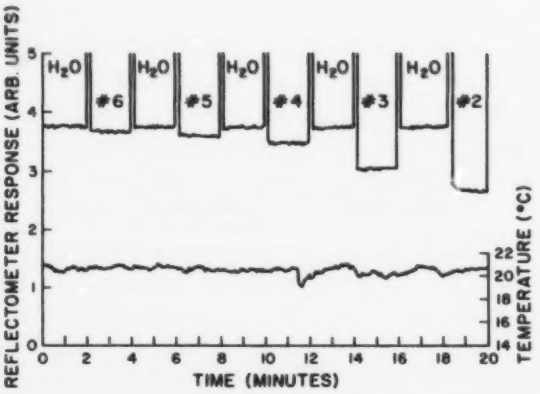

Figure 7 Response of single wavelength (840 nm) reflectometer to various concentrations (samples 2, 3, 4, 5, 6 - Table 1) of urea in water. The probe tip was immersed in distilled water between each immersion in a test solution. Amplifier time constant was 1.25 ms.

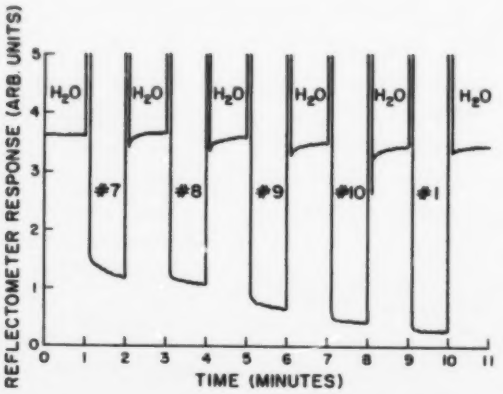

Figure 8 Response of single wavelength (840 nm) reflectometer to various concentrations (samples 1, 7, 8, 9, 10 - Table 1) of urea in water. The probe tip was immersed in distilled water between each immersion in a test solution. Amplifier time constant was 1.25 ms.

shown that the best method of obtaining a clean fibre tip is by cleaving the fibre. This was **not** done in this instance. The only problem posed by these experiments was due to the presence of small particles on the liquid surface of the limited samples provided. These particles, many of which appeared to be generated each time a vial cap was removed, had the potential of interfering with the experiment, each time the fibre was immersed in a solution.

SUMMARY

It was shown that the fibre reflectometer had the sensitivity required to monitor the presence of urea in water even in the case of very small concentrations. Both experimental units tested were able to distinguish the samples on the basis of the optical signals. Even in the case of the reflectometer for which some details of the response were not explained with certainty, it can be said that the relative responses to the samples were in accord with the results obtained with the other unit. It is thought that the anomalies were probably caused by contamination of the sensing tip. Nonetheless, a reasonably complete discussion of these anomalies was included in the hope that another explanation, perhaps based on some characteristics of the samples unknown to me, might be suggested.

Further trials would be needed to more fully explore the suitability of the fibre reflectometer (refractometer) for the task of monitoring a changing urea concentration over an extended period of time.

REFERENCE

1. Legendre, J.P. A simple reflectometer configured to sense temperature. Proc. SPIE, "Fiber Optic and Laser Sensors III", 1985. Vol. 566, pp. 321-326.